Thursday Morning, October 23, 2008

Tribology Focus Topic
Room: 205 - Session TR+SE+TF-ThM

Advances in Surface Engineering for Friction and Wear Control
Moderator: K.J. Wahl, U.S. Naval Research Laboratory

8:00am **TR+SE+TF-ThM1 Evaluation of Ti-In-N Films for Tribological Applications**, ***J.E. Krzanowski***, *M. Nowicki*, University of New Hampshire

Titanium nitride and indium have both found applications as tribological coatings. While TiN is used primarily for its high hardness and wear resistance, indium can be used as a solid lubricant layer. In this study, we have examined the concept of using co-deposited TiN-In films for tribological applications. Ti-In-N films have been deposited by RF co-sputtering of Ti and In in a nitrogen/argon atmosphere. By varying the power to the Ti and In sources, the In/Ti ratio in the film was varied. Films were deposited at DC substrate bias levels of -50V and -150V. In both cases, as the In/Ti power ratio was increased, the indium content increased, but in a highly non-linear manner. At lower power ratios, the films had a cubic TiN structure, but as the power ratio increased, there was an abrupt transition to a hexagonal structure. Near the transition point, the films could also be amorphous, depending on film thickness, and thicker films were more likely to be crystalline. Below the transition point, the film composition depended strongly on substrate bias, and films deposited at -150V bias exhibited significantly reduced indium contents. Tribolgical tests were conducted using a pin-on-disk test with an alumina counterface. Most films showed shorter wear lives compared to TiN alone. The friction coefficients were found to depend on the In content in the films, and it was also found that heating films for short times in the range of 100-250C reduced friction coefficients.

8:20am **TR+SE+TF-ThM2 Nanostructured Sulfur Doped CHx-TiB2 Coatings for Improved Mechnical and Friction Performance**, ***B. Zhao***, *Y.W. Chung*, Northwestern University

Hydrogenated amorphous carbon films are known to attain ultra-low friction performance only in dry environments. Our work demonstrated that sulfur doping of hydrogenated carbon films results in ultra-low friction performance in both dry and humid environments. However, these films have a hardness of 7 - 10 GPa and an elastic modulus around 80 GPa, which are too low for some high stress applications. Formation of nanostructured coatings is known to improve hardness. With the aim to produce hard, low-friction coatings, we synthesized nanolayered and nanocomposite films of sulfur-doped hydrogenated carbon and titanium diboride using dual-target magnetron sputtering. Titanium diboride deposited by this method had a hardness >30 GPa. This paper will discuss the film structure and how such structure correlates with its tribological and mechanical properties.

8:40am **TR+SE+TF-ThM3 Latest Developments on the Family of C-alloyed TMD Self-lubricating Coatings**, ***A. Cavaleiro***, SEG-CEMUC - University of Coimbra, Portugal, *T. Polcar*, CTU Prague, Czech Republik, *M. Evaristo*, SEG-CEMUC - University of Coimbra, Portugal **INVITED**

Due to their layered structure and weak inter-layer bonding, transition metal dichalcogenides (TMD) exhibit very interesting physical and tribological properties. Among different TMD families (TmS2, TmSe2 and TmTe2, with Tm = Mo, W, Nb) MoS2 and WS2 have been the most intensively studied in last decades. They are now currently used either as oil additives or as thin self-lubricating coatings. The industrial applicability of these coatings is still very limited owing to their two main drawbacks: (1) the loss of the tribological performance in humidity-containing environments and, (2) the low load bearing capacity. Several solutions have been proposed to overcome these problems as, for example, alloying TMD coatings with other elements or compounds, such as C, Ti, Pb, and TiN. The concept of coatings based on TMDs alloyed with carbon was introduced in the 90´s and was based on the expected synergy between the excellent frictional behavior of TMD in vacuum/dry air and the tribological performance of C-based materials. Improved frictional performance of the W-S-C coatings could be achieved when the coatings were tested by environmental cycling from dry to humid air (FC - friction coefficient from 0.02 to 0.15). Successive changes in the sliding mechanisms based on the modification of the contact layers were assigned as responsible for this behaviour. The TMD+C solution was adopted by the authors for their research by studying its extension to other TMD-C systems. The aim of this talk was to present the latest developments achieved within TMD-C magnetron sputtering deposited coatings concerning di-selenides (Mo-Se-C and W-Se-C). Lower friction coefficient was achieved in comparison to previous deposited W-S-C coatings, particularly in humid air. FC was possible to be kept lower than 0.05 in all testing conditions. Tests were performed with contact stress as high as 1.5 GPa without destruction of the coating. The frictional and wear mechanisms under different operating conditions were studied by nanoscale analysis of the wear tracks. The tribological performance, whatever the testing conditions, was attributed to the formation of a thin tribolayer consisting exclusively of TMD platelets with the (002) plans oriented parallel to the sliding motion. C is removed from the contact area during the re-orientaion process, only playing a secondary role by increasing the coatings density, avoiding surface oxidation and improving the loading bearing capacity.

9:20am **TR+SE+TF-ThM5 Reduction in Friction and Micropitting by Coatings and Lubricants Containing Inorganic Fullerenes**, ***S.J. Bull***, *A. Oila*, Newcastle University, UK

Improvement in component performance by reduction in friction and wear has been the focus of considerable research over the last forty years. As products become more highly engineered and component size is reduced the significance of friction and wear is increased, particularly in terms of improvements in energy efficiency, and the need to develop materials with improved triboloigcal performance becomes critical. Nanostructured materials are one way whereby this might be achieved. Recent work has focussed on the development and assessment of nanomaterials and composites for triboloigcal performance. In particular, the use of inorganic fullerene-like materials in the form of coatings and nanoparticles (and as the reinforcement for nanocomposites) has shown a lot of promise for tribological applications. This talk will highlight the use of inorganic fullerene nanoparticles as additives for lubricating oils or coatings to achieve a significant increase in the operational life of rolling/sliding components such as gears.

9:40am **TR+SE+TF-ThM6 Tribology of Carbon Films in Hydrogen and Deuterium Gas Environments**, *A. Erdemir*, ***O.L. Eryilmaz***, Argonne National Laboratory

Recent systematic studies in our laboratory have shown that certain diamond-like carbon (DLC) films are able to achieve superlow friction and wear when tested in hydrogen-containing test environments. In the presence of deuterium, we were also able to achieve very low friction and wear on these films. In this study, we used a combination of controlled-environment atmospheric pressure and vacuum tribometers to further verify the critical effects of hydrogen and deuterium on friction and wear of such films; then we used imaging SIMS and XPS methods to ascertain the near surface chemistry of their sliding surfaces. The combined results of tribological tests and surface analytical studies revealed that there exist a close correlation between the chemical nature of sliding DLC surfaces and their friction and wear behaviour. Specifically, we found that in the presence of both hydrogen and deuterium, the sliding contact areas of carbon films were covered by a hydrogen and deuterium film (only a few Å thick). The wear rates and friction coefficients are much higher if tests were run in dry nitrogen or vacuum than in hydrogen and deuterium. Overall, we show that superlow friction behaviour of certain DLC films is largely controlled by gas-surface interactions.

10:40am **TR+SE+TF-ThM9 Tribomaterials for Spacecraft: Testing & Surface Chemistry**, ***J.R. Lince***, The Aerospace Corporation **INVITED**

The spacecraft environment is challenging for tribocoatings and lubricants used in devices in satellites and launch vehicles. Areas of concern include vibration during launch, thermal cycling on orbit, and the need to work effectively for missions up to twenty years in duration without lubricant replenishment. Especially challenging is the need for tribomaterials to withstand the vacuum of space during lengthy missions. Spacecraft tribomaterials must exhibit low vapor pressures, since evaporation of lubricants can result in loss from and premature failure of devices, as well as contamination of sensitive spacecraft components. A relatively small set of liquid lubricants meet the vapor pressure requirement – while also meeting performance requirements for current spacecraft applications – including synthetic hydrocarbons and perfluorinated polyalkylethers. Soft solid lubricants such as molybdenum disulfide (MoS_2) and polytetrafluoroethylene (PTFE) have been used traditionally. More recently, hard low friction coatings such as hydrogenated diamond-like carbon have shown promise for operation in vacuum with existing spacecraft lubricants, or even unlubricated operation in vacuum. These solid- and liquid-based tribomaterials show performance in vacuum that differs with that in air, nitrogen, or even with small partial pressures of oxygen and water. This is especially important for spacecraft hardware, because it is often prohibitive to test them in a space-like environment, including vacuum, before launch.

As such, differences between non-vacuum and vacuum testing need to be understood in order to predict how lubricated devices will perform in space. This talk will focus on a series of recent studies done at The Aerospace Corporation that elucidate the effects of vacuum and other environments on the tribological performance of several important spacecraft tribomaterials. The emphasis will be on how varying environments affect the surface chemistry of the materials.

11:20am **TR+SE+TF-ThM11 Nano-Smooth Diamond Coatings on Various Alloys for Ultralow Friction in the Presence of OH-Containing Lubricants**, ***T. Gries***, CNRS - ICARE, France, *C. Matta, M.I. De Barros Bouchet, B. Vacher,* Ecole Centrale de Lyon, France, *S. de Persis,* ICARE - CNRS, France, *L. Vandenbulcke*, CNRS, France

Titanium alloys and titanium-coated alloys are important materials for aerospace, mechanical, chemical and biomedical applications; however their applications could be extended by improving their tribological behaviour. This can be done by using diamond-based coatings which are outstanding materials for changing their surface properties. We have shown that nano-smooth fine-grained diamond coatings could be deposited on these alloys at moderate temperature, equal to or lower than 600°C, from CH_4-CO_2 species. They are in fact duplex coatings with an external diamond film, a titanium carbide sub-layer and a diffusion solid-solution. These coatings exhibit particularly strong adherence with the substrates as shown by various mechanical tests and very high induced stresses without peeling off. They are first described in terms of sp^2-hybridized carbon contents relatively to the sp^3-carbon ones, a parameter which influences the structure and the intrinsic diamond properties (surface roughness in the 15-35 nm range, micro-hardness, Young's modulus and residual stresses). The whole is correlated to the plasma enhanced CVD process through the formation of different concentrations of the gaseous precursors in the plasma which include both radicals and stable species as revealed by molecular beam mass spectrometry and corroborated by kinetic calculations in the C-H-O plasmas. These coatings are studied by micro-Raman spectroscopy and their structure is revealed by TEM studies. A sp^2-C enriched layer is especially evidenced at their extreme surface by Energy-Filtered TEM on transverse cross-sections, a layer which is important for tribochemical reactions. While the friction coefficient is high under ultra high vacuum, ultra low friction is obtained in saline corrosive solution. Ultra low friction with no wear is also obtained with gas phase lubrication by glycerol under boundary lubrication regime, in conditions which permit a better identification of the friction mechanism from advanced surface characterizations. These studies allow concluding that lubrication of these diamond coatings by OH-containing molecules can permit new or improved applications in various fields. Some examples of ultra low friction and low wear are provided when nano-smooth diamond coatings or alumina are sliding on nano-smooth diamond in corrosive saline solution or in the presence of glycerol lubricant, a model of environmentally friendly molecules.

11:40am **TR+SE+TF-ThM12 Tribology of Nanocrystalline Diamond Coatings**, ***N.D. Theodore***, North Carolina State University, *K.J. Wahl*, Naval Research Laboratory

The tribological behavior of several nanocrystalline diamond (NCD) coatings was compared to correlate compositional, structural, and chemical bonding differences to their friction performance. The diamond coatings were confirmed by X-ray diffraction (XRD) to be nanocrystalline with crystallite sizes ranging from to 4 to 60 nm. These diamond coatings could be differentiated from each other by their visible wavelength Raman absorption bands. Some coatings had a single strong peak at 1332 cm^{-1} typical of crystalline diamond bonding; others also had broad peaks at 1340 cm^{-1} and 1580 cm^{-1} characteristic of the D and G peaks in sp^2 hybridized carbon; and still others had additional peaks at 1135 cm^{-1} and 1470 cm^{-1}, which are commonly attributed to polyacetylene bonding. Reciprocating sliding tests using diamond counterfaces in controlled humidity environments resulted in low friction values for all coatings, between 0.02 and 0.09. The coatings exhibiting lower friction values possessed lower mean surface roughnesses, as measured using an atomic force microscope (AFM), smaller crystallite sizes, and increased amounts of non-sp^3 carbon content. Transmission Fourier transform infrared (FTIR) microscopy was used to examine the bonding chemistry in the coatings and wear tracks. The role of diamond coating microstructure, surface roughness, and bonding chemistry to the tribological behavior of NCD will be presented.

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